



Effect of Aging on Stained Monolithic Resin-Ceramic CAD/CAM Materials: Quantitative and Qualitative Analysis of Surface Roughness

Mühlemann, Sven ; Bernini, Joëlle M ; Sener, Beatrice ; Hämmerle, Christoph H F ; Özcan, Mutlu

Abstract: **PURPOSE** The aim of this in vitro study was to measure the effect of staining and artificial aging on the surface roughness of commercially available resin-ceramic computer-aided design and computer-aided manufacturing (CAD/CAM) materials both quantitatively and qualitatively and to compare it to feldspathic material. **MATERIALS AND METHODS** Test specimens (n = 15 per material) were prepared of CAD/CAM ingots from a resin nanoceramic (Lava Ultimate, LVU), a polymer-infiltrated ceramic (Vita Enamic, ENA), and a resin nanoceramic (Cerasmart, CER). In the staining protocol, test specimens were (i) roughened in a standardized manner and (ii) stained with the manufacturer's recommended staining kit by means of photo-polymerization (Bluephase Polywave). The control specimens were prepared out of a feldspathic ceramic (Vita Mark II, VM2) and stained in a ceramic furnace. As negative control of each group, 15 specimens were prepared and polished in a standardized manner. Surface roughness (Ra) was measured after finishing procedures and after simulation of clinical service up to 5 years by means of toothbrushing. After each year of aging, one specimen per group was randomly selected for scanning electron microscopy (SEM) analysis. Kruskal-Wallis test and paired post-hoc test were applied to detect differences between treatment groups ($\alpha = 0.05$). **RESULTS** The mean roughness measurements of the stained CAD/CAM materials were $0.14 \pm 0.04 \mu\text{m}$ (ENA), $0.15 \pm 0.03 \mu\text{m}$ (LVU), $0.22 \pm 0.03 \mu\text{m}$ (VM2), and $0.26 \pm 0.12 \mu\text{m}$ (CER). In the polished CAD/CAM materials the measurements were $0.01 \pm 0.01 \mu\text{m}$ (CER), $0.02 \pm 0.01 \mu\text{m}$ (LVU), $0.02 \pm 0.00 \mu\text{m}$ (VM2), and $0.03 \pm 0.01 \mu\text{m}$ (ENA). Irrespective of the restoration material, the applied staining protocol resulted in a higher surface roughness compared to the polished specimens ($p < 0.001$). After 5 years of simulated aging the mean surface roughness in the stained CAD/CAM materials were $0.22 \pm 0.03 \mu\text{m}$ (VM2), $0.24 \pm 0.09 \mu\text{m}$ (ENA), $0.25 \pm 0.06 \mu\text{m}$ (CER), and $0.37 \pm 0.09 \mu\text{m}$ (LVU). Aging had a significant effect on surface roughness in groups ENA and LVU ($p < 0.001$). SEM analysis showed that the staining layer on resin-ceramic CAD/CAM materials was partially removed over time. **CONCLUSIONS** The applied staining protocol significantly increased surface roughness of CAD/CAM materials. Instability of the staining layer on resin-ceramic CAD/CAM materials could be anticipated over time as a consequence of toothbrushing, whereas feldspathic ceramic did not suffer from such aging effect.

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**Effect of aging on stained monolithic resin-ceramic CAD-CAM materials:
quantitative and qualitative analysis of surface roughness**

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Running head: Surface roughness of stained resin-ceramic materials

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Abstract

Purpose: The aim of this in vitro study was to measure the effect of staining and artificial aging on the surface roughness of commercially available resin-ceramic Computer aided design and computer aided manufacturing (CAD-CAM) materials both quantitatively and qualitatively and to compare it to feldspathic material.

Materials and methods: Test specimens (n=15 per material) were prepared out of CAD-CAM ingots from a resin nanoceramic (LVU; Lava Ultimate; 3M ESPE), a polymer-infiltrated ceramic (ENA; Vita Enamic; Vita Zahnfabrik) and a resin nanoceramic (CRS; Cerasmart, GC Corporation). In the staining protocol, test specimens were i) roughened in a standardized manner and ii) stained with the manufacturer's recommended staining kit by means of photopolymerization (Bluephase Polywave; Ivoclar Vivadent). The control specimens were prepared out of a feldspathic ceramic (VM2; Vita Mark II; Vita Zahnfabrik) and stained in a ceramic furnace. As negative control of each group, 15 specimens were prepared and polished in a standardized manner. Surface roughness (Ra) was measured after finishing procedures and after simulation of clinical service up to five years by means of toothbrushing. After each year of aging one specimen per group was randomly selected for Scanning Electron Microscopy (SEM) analysis. Kruskal-Wallis test and paired Post-hoc test were applied to detect differences between treatment groups ($\alpha=0.05$).

Results: The mean roughness measurements of the stained CAD-CAM materials were $0.14 \pm 0.04 \mu\text{m}$ (ENA), $0.15 \pm 0.03 \mu\text{m}$ (LVU), $0.22 \pm 0.03 \mu\text{m}$ (VM2), and $0.26 \pm 0.12 \mu\text{m}$ (CER). In the polished CAD-CAM materials the measurements were $0.01 \pm 0.01 \mu\text{m}$ (CER), $0.02 \pm 0.01 \mu\text{m}$ (LVU), $0.02 \pm 0.00 \mu\text{m}$ (VM2), and $0.03 \pm 0.01 \mu\text{m}$ (ENA). Irrespective of the restoration material, the applied staining protocol resulted in a higher surface roughness compared to the polished specimens ($p < 0.001$). After 5 years of simulated aging the mean surface roughness in the stained CAD-CAM materials were $0.22 \pm 0.03 \mu\text{m}$ (VM2), $0.24 \pm 0.09 \mu\text{m}$ (ENA), $0.25 \pm 0.06 \mu\text{m}$ (CER), and $0.37 \pm 0.09 \mu\text{m}$ (LVU). Aging had a significant effect on surface

roughness in groups ENA and LVU ($p < 0,001$). SEM analysis showed that the staining layer on resin-ceramic CAD-CAM materials was partially removed over time.

Conclusions: The applied staining protocol significantly increased surface roughness of CAD-CAM materials. Instability of the staining layer on resin-ceramic CAD-CAM materials could be anticipated over time as a consequence of toothbrushing, whereas feldspathic ceramic did not suffer from such aging effect.

Keywords: staining, resin-ceramic, CAD, CAM, monolithic, roughness, REM, Ra

Introduction

CAD-CAM technology was introduced in dentistry in the late 1990s. Since then, primarily ceramic materials were processed using CAD-CAM¹. Today, various materials including resin composite and hybrid resin-ceramic materials are available for digital manufacturing processes². Resin-ceramic materials were recently introduced to the market and aim to combine the advantages of both restoration materials, resin and ceramic restoration materials³⁻⁶.

CAD-CAM technology enables to increase treatment time efficiency for the patient in a so-called chairside workflow⁷. Intraoral scanners allow dentists to digitize the intraoral situation at the time of impression and to process the digital data in an associated CAD-CAM system for the fabrication of a dental restoration. Thereafter, the restoration may be cemented at the same appointment. Generally, these restorations are milled out of CAD-CAM materials in an anatomical full-contour. The monolithic design allows to avoid any further processing by the dental technician, which is detrimental regarding time efficiency⁸. The finishing process is limited to polishing procedures to reduce the rather high initial surface roughness created by the rotary instruments of the milling machine. An in-vitro study showed that polishability and behavior against artificial toothbrushing of resin ceramic CAD-CAM materials depended on specific micromechanical properties⁹. In addition, when polishing discs were used significantly lower surface roughness was obtained compared to silicone polishers².

CAD-CAM materials for chairside manufacturing are available in different colors, as well as with color gradients through which the natural appearance of a tooth can be simulated. However, monolithic restorations without further processing do not meet high aesthetic demands and therefore, are more often used in posterior non-aesthetic sites. For monolithic ceramic restorations stain firing is available in order to overcome this aesthetic limitation. In contrast, firing processes for resin-ceramic CAD-CAM materials containing acrylate polymer matrix are contraindicated.

Manufacturers of resin-ceramic CAD-CAM materials offer staining kits to meet higher aesthetic demands. Generally, the application process involves a chemical conditioning of the surface and photo polymerization of the staining material. The procedure can be executed chairside and does not involve a dental technician and further equipment. However, information on the effect of staining to the surface roughness is unknown.

Surface roughness is an important factor for dental restorations in terms of aesthetic, technical and biological success which could also negatively affect the translucency of a restoration¹⁰. In ceramic restorations, the risk for crack initiation and propagation was reported to be higher when the surface was rougher¹¹. In addition, it was shown, that the wear rate of an antagonist is highly influenced by the surface roughness of the opposing restoration¹². Finally, surface roughness has an influence on bacterial adhesion. A higher surface roughness led to a higher amount of plaque accumulation and consequently the risk of gingivitis, secondary caries, and discoloration may be higher¹³⁻¹⁶. A review postulated that the critical surface roughness for increased bacterial adhesion is 0.2 micrometers¹⁷.

The oral environment may induce alterations to any restoration material due to mechanical stress through mastication and the use of cleaning devices. Wear of tooth substance and restoration material is a common clinical phenomenon. It was demonstrated, that the abrasiveness of a tooth paste and the type of restoration may have an influence on the amount of wear^{18,19}. An in-vitro study demonstrated, that resin-ceramic CAD-CAM materials showed similar wear behavior to natural enamel²⁰. However, in this study the CAD-CAM materials were investigated without staining.

Today, a large variety of different resin-ceramic CAD-CAM materials exist for the fabrication of monolithic restorations. Yet, no scientific data is available on the effect of staining procedures and aging to the surface roughness of resin-ceramic CAD-CAM materials. Therefore, the purpose of the present in-vitro study was to evaluate the surface roughness of stained and polished resin-ceramic CAD-CAM materials over time. The null hypotheses were

that there would be no difference in surface roughness between stained and polished resin-ceramic CAD-CAM materials and that aging would have no influence on the surface roughness.

Materials and Methods

Feldspathic ceramic (VM2; Vita Mark II; Vita Zahnfabrik) as the control, a resin nanoceramic (LVU; Lava Ultimate; 3M ESPE), a polymer-infiltrated ceramic (ENA; Vita Enamic; Vita Zahnfabrik) and a resin nanoceramic (CRS; Cerasmart, GC Corporation) were tested in this study. The chemical composition of each CAD-CAM material is listed in Table 1. Thirty specimens ($12 \times 14 \times 2.5 \text{ mm}^3$) were cut from each CAD-CAM ingot ($12 \times 14 \times 18 \text{ mm}^3$) with a saw microtome (SP1600, Leica Microsystem). Standardized thickness of 2.5 millimeters was checked mechanically with a thickness gauge. The circumference of each specimen was manually rounded by means of a diamond bur (879L/FG314, Torpedo Lang) in order to prevent damage to the brushes during aging. The specimens were adhesively fixed (ScandiQuick, SCAN DIA) to the specimen carrier (SEM carrier, Carl Zeiss).

One half ($n=15$) of the ground specimens was polished under water-cooling in a standardized manner using a polishing machine (Planapol 2; Struers) at 300 revolutions per minute and 3 consecutive silicon carbide papers for 15 seconds each (P1200, P2500, P4000 Microcut; Buehler). Specimens were manually finished using polishing felt (12 Microclouth PSA; Buehler) and a polishing suspension (MasterMet 0.05 μm ; Buehler).

The other half ($n=15$) had to undergo a staining protocol. First, specimens were roughened in a standardized manner using a grinding machine (Planapol; Struers) at 300 revolutions per minute and 15 μm abrasive paper (custom-made; Intensiv) for 15 seconds. Thereafter, the surface of all specimens was air-borne particle abraded with 50 μm aluminium oxide (1 bar at 1 centimeter distance for 10 seconds; Unitool AG). Staining procedures were applied according to the manufacturer's instructions. The composition and the application procedure of each staining kit is listed in Table 2. Polymerization was executed in a

standardized sequence with light curing by means of a curing hand piece (Bluephase Polywave 380-515nm; Ivoclar Vivadent) for 90 seconds and by means of a laboratory curing device (ESPE VISIO BETA Vario, program 1) for 15 minutes under vacuum. In the control group, feldspathic ceramic staining was applied in a two-step procedure with stain and glaze firing (Program 2, Austromat D4 X; Dekema)

Artificial toothbrushing was simulated in a custom-made device (ZMB 8, University of Zurich) using a toothbrush (PARO M43; Esro) at a standardized pressure of 2.5 Newton in a toothpaste slurry containing a tooth paste of 100 RDA (Radioactive Dentin Abrasion)²¹. Toothbrushes and toothpaste slurry were changed every 3 hours and every 48 hours, respectively. One year of clinical service was simulated by 3650 cycles (7300 toothbrushing strokes) assuming that a tooth surface is brushed 20 times per day²². In total, 5 years of clinical service were simulated.

The surface roughness of each specimen was analyzed after finishing procedures and after each year of aging with a profilometer (Form Talysurf 50; Taylor Hobson) by measuring the average surface roughness (Ra in μm). Standardization was achieved by following ISO 4288:1998. Five measurements per specimen were made in both the lateral and longitudinal direction and a mean Ra value was calculated.

For the qualitative characterization of aging patterns specimens were visually examined by scanning electron microscopy (SEM) (Carl Zeiss Supra 50VP FESEM; Carl Zeiss). Before and after each year of aging, one specimen of each group was randomly selected for imaging. The specimens were dried and sputtered with gold (Sputter SCD 030; Baltec) 24 hours before the analysis. Images of 50, 200, 1000, and 5000 times magnification were made. After SEM imaging, specimens were not available for further aging. Therefore, the number of specimens was reduced by one after each year of aging.

Data was coded in Excel and statistical analysis were performed with the statistical software R (R Foundation for Statistical Computing; www.R-project.org), with the package

Pairwise Multiple Comparison of Mean Ranks Package (PMCMR)²³ for pairwise posthoc comparisons using rank s, and with the package ggplot2²⁴ for elegant graphical outputs. Continuous variables were reported by using means and ranges. Differences between material groups were calculated using Kruskal-Wallis test followed by Wilcoxon signed rank test. Resulting P values were corrected with the Holm adjustment for multiple testing. Differences within material groups before and after aging were analyzed by Wilcoxon signed rank test. The level of significance was set at $\alpha = 0.05$ and accordingly 95% confidence intervals

Results

The mean Ra of the stained and polished CAD-CAM materials after finishing procedures and after each year of aging is shown in Table 3 and illustrated in Figure 1. For all CAD-CAM material irrespective of the time point, Ra was significantly higher in the stained group as compared to the polished group ($p < 0,001$).

Within the stained CAD-CAM materials, mean roughness measurements after finishing procedures were $0.14 \pm 0.04 \mu\text{m}$ (ENA), $0.15 \pm 0.03 \mu\text{m}$ (LVU), $0.22 \pm 0.03 \mu\text{m}$ (VM2), and $0.26 \pm 0.12 \mu\text{m}$ (CER). Group CER showed significantly higher surface roughness than all other resin-ceramic CAD-CAM materials (ENA $p < 0,001$, LVA $p < 0,001$). Ra of group ENA and group LVA was significantly lower compared to the control group VM2 ($p < 0,001$).

After 5 years of aging, the mean roughness measurements were $0,22 \pm 0,03 \mu\text{m}$ (VM2), $0,24 \pm 0,09 \mu\text{m}$ (ENA), $0,25 \pm 0,06 \mu\text{m}$ (CER), and $0,37 \pm 0,09 \mu\text{m}$ (LVU). The increase of Ra was statistically significant in group ENA ($p < 0,001$, 95% CI -0,14; -0,06) and in group LVA ($p < 0,001$, 95% CI -0,28; -0,15). The mean roughness in group CER decreased with aging and increased after 5 years with no significant difference before aging ($p = 0,64$, 95% CI -0,032; 0,059). In control group VM2, aging did not show a significant effect ($p = 0,32$, 95% CI -0,005; 0,013).

Within the polished CAD-CAM materials, the surface roughness before aging were $0,01 \pm 0,01 \mu\text{m}$ (CER), $0,02 \pm 0,01 \mu\text{m}$ (LVU), $0,02 \pm 0,00 \mu\text{m}$ (VM2), and $0,03 \pm 0,01 \mu\text{m}$ (ENA). The statistical analysis showed the following general ranking of Ra from lowest Ra to highest Ra: group CER < group LVU = group VM2 < group ENA ($p < 0,001$). After 5 years of aging, a statistically significant increase of mean surface roughness was calculated ($p < 0,001$). The values were $0,03 \pm 0,01 \mu\text{m}$ (CER), $0,04 \pm 0,00 \mu\text{m}$ (ENA), $0,04 \pm 0,01 \mu\text{m}$ (VM2), and $0,06 \pm 0,01 \mu\text{m}$ (LVU).

SEM images at a magnification of 5000 showing the center of the specimens are presented in Figures 2-5. The surface of stained specimens showed a rather smooth and homogenous surface in groups CER and VM2, whereas in group ENA voids were present and in group LVU irregularities were observed on the specimen surfaces. In the stained specimens of the resin ceramic CAD-CAM materials a partial loss of the staining layer after 1 year of aging with a smoothing of the borders by further aging was observed. In the stained control group VM2 specimens demonstrated no loss of surface integrity. Generally, visible changes in the polished specimens were limited. However, in group LVU an inhomogeneous surface structure was already present after 1 year of aging as compared to the image before aging. In group ENA the surface was already inhomogeneous before aging and seemed to be smoothed by means of toothbrushing.

Discussion

The present study showed that the applied staining protocol significantly increased surface roughness of resin-ceramic CAD-CAM materials. Thus, the first null hypothesis was rejected. In addition, aging by means of toothbrushing significantly increased surface roughness of stained resin-ceramic CAD-CAM materials, whereas stained ceramic CAD-CAM material did not show significant changes of surface roughness. Therefore, the second null-hypothesis was also rejected.

The results of this study showed that the applied staining protocol increased surface roughness irrespective of the CAD-CAM material used. The differences in mean surface roughness between the CAD-CAM materials were statistically significant. However, a standardized surface roughness of CAD-CAM material after finishing procedures was not possible to achieve due to the manual application process as well as the different composition of the staining materials. Among the resin ceramic CAD-CAM materials, with a resin nano ceramic (LVA) and a polymer-infiltrated ceramic (ENA) and the corresponding staining kit, the lowest surface roughness was achieved.

It was postulated that the critical surface roughness for increased bacterial adhesion is 0.2 micrometers¹⁷. Consequently, the risk of gingivitis, secondary caries, and discoloration may be higher¹³⁻¹⁶. In one test group (CER) and with feldspathic ceramic, staining resulted in a mean surface roughness above this threshold. While staining of resin ceramic CAD-CAM materials is a single step procedure, staining of monolithic ceramic materials is normally a multiple step application and firing process. In the present study, feldspathic ceramic staining was limited to a two-step procedure with stain and a glaze firing. It was previously demonstrated that surface roughness of ceramic specimens fired 10 times was significantly lower than that of the same specimens fired twice²⁵. Staining of monolithic ceramic materials is therefore a time consuming procedure and a minimally rough surface may be achieved⁸.

Based on these results of the present study, the applied staining protocol of resin ceramic CAD-CAM materials may not be recommended. Aging had a serious effect on the surface roughness of stained resin ceramic CAD-CAM materials. In two resin-ceramic materials (ENA, LVA), a significantly higher surface roughness was detected after 5 years of simulated aging. In group CER, aging resulted in a polishing effect within 2 years and was followed by a roughening effect. In contrast, surface roughness in the stained feldspathic ceramic material was constant over the simulated time. The results of the present study are in accordance with a recently published study demonstrating that for surface roughness in stained leucite-based

ceramic no statistically significant difference was found after 12 years of toothbrushing²⁶. In contrast, a clinical study showed that glaze layers were worn after 6 months²⁷, which may require polishing of the surfaces after glazing²⁸.

The SEM pictures showed a partial loss of the staining layer in the stained resin ceramic CAD-CAM materials. A polishing effect by toothbrushing could be observed at the borders of areas, where the staining layer was lost. It was demonstrated, therefore, that the calculated mean surface roughness always has to be interpreted in relation to the corresponding SEM pictures.

In the present study, a common parameter for roughness measurements was chosen, namely average roughness (Ra). It might be hypothesized that partial loss of the staining layer would have been detected with the parameter roughness depth (Rz), which is more influenced by extremes. However, an in-vitro study evaluating the effect of artificial toothbrushing on surface roughness of CAD-CAM materials could not demonstrate an added value by applying both parameters, Ra and Rz⁹.

The roughness of restoration materials may have an effect on the wear rate of the antagonist. An in-vitro study demonstrated that the glaze layer of zirconia restorations was removed after 120'000 chewing cycles and in consequence resulted in a high wear rate of the antagonist¹². Loss of glaze layer had exposed the pretreated surface, which was air-borne particle abraded and presented a higher roughness. Similarly, loss of staining layer in the resin-ceramic materials could have the same effect as the surface was pretreated by air-borne particle abrasion as well. However, wear rate was not evaluated in the present study.

Polished resin-ceramic CAD-CAM materials showed significantly lower surface roughness as compared to the stained resin-ceramic materials. The mean values were below the postulated critical surface roughness of 0.2 micrometers¹⁷ after finishing procedures and after 5 years of aging ranging between 0.03 μm and 0.06 μm . Therefore, polished resin-ceramic CAD-CAM materials can be recommended for clinical use without application of a staining layer. However, significant differences were calculated before and after 5 years of aging. The

results of the present study confirm an in-vitro study showing that abrasive toothbrushing significantly increased roughness of resin ceramic CAD-CAM materials²⁰. SEM images showed structural changes of the surface in the resin ceramic CAD-CAM materials. It may be speculated, that the resin matrix has been worn out by aging. A previous in-vitro study showed that CAD-CAM materials with low surface hardness such as resin-ceramic materials are more prone to degradation by artificial tooth brushing⁹. It is, however, unknown what effect these changes would have on the material properties and the clinical performance. Therefore, the results of the present study require clinical verification. Yet, in vitro studies allow a comparative evaluation of different materials under standardized conditions.

Conclusion

From this study, the following could be concluded:

- Regardless of the resin-ceramic CAD-CAM material, the applied staining protocol significantly increased the surface roughness.
- Except for stained feldspathic ceramic CAD-CAM, simulated aging by means of toothbrushing significantly increased surface roughness of stained resin-ceramic CAD-CAM materials.

Captions to the tables and legends:

TABLES

Table 1. Chemical composition of CAD-CAM materials.

Table 2. Chemical composition of staining materials.

Table 3. Surface roughness (Ra) of 4 different resin-ceramic CAD-CAM materials (n=10). CER, Cerasmart (GC Corporation); ENA, Vita Enamic, Vita Zahnfabrik; LVU, Lava Ultimate (3M Espe); VM2 (Vita Mark 2, Vita Zahnfabrik).

LEGENDS

Fig. 1. Surface roughness (Ra) of 4 different resin-ceramic CAD-CAM materials (n=10).

Fig. 2. SEM pictures group CER (5000 x magnification). Note that partial loss of staining layer is evident (b-f).

Fig. 3. SEM pictures group ENA (5000 x magnification). Note that partial loss of staining layer is evident (b-f) and that surface roughness decreased after exposure to aging in the polished specimens (g compared m).

Fig. 4. SEM pictures group LVU (5000 x magnification). Note that (*) are probably remnants of the slurry remained attached on the rough zones which were detached after thorough cleaning. In the polished specimens h-m a loss of acrylate polymer matrix is present.

Fig. 5. SEM pictures group VM2 (5000 x magnification). Note the stability of the staining layer over time (a-f).

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